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The research is part of a several year study of the electron stimulated desorption (ESD) behavior of transition metal oxides. The original objective of this research was the development and implementation of an ESD O atom source to be used in Low Earth Orbital (LEO) simulations of materials damage. Though the desorption of neutral particles has been widely postulated, most of the ESD results found in the literature are measurements of ejected ions. The principal reason for this is the ease of ion detection and the inherently low sensitivity of detectors to neutral particles. Most information pertaining to the relative abundance of neutral versus ion desorption is the result of indirect measurements. Despite extensive experimentation including the application of resonant enhanced multiphoton ionization for the selective detection of atomic oxygen the production of atomic oxygen via ESD was never detected. From careful calibration experiments the upper limit to the neutral to ion yield, O/O^+, by ESD was calculated to be 10. From electron microscopy studies of both the reconstructed Ir(001) and Si(001) surfaces, the controversy between the existing models in literature (±0.016 nm for the Ir(001)-5x1 models and ±0.005 nm for the Si(001)-2x1 models) could be resolved for the first time using a combination of UHV-TED and UHV-HREM techniques.

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EXPERIMENTAL INVESTIGATIONS AND MOLECULAR DYNAMICS

SIMULATIONS OF RADIATION INDUCED SURFACE DAMAGE

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Abstract

This research is part of a several year study of the electron stimulated desorption (ESD) behavior of transition metal oxides. The original objective of this research was the development and implementation of an ESD O atom source to be used in Low Earth Orbital (LEO) simulations of materials damage. Though the desorption of neutral particles has been widely postulated, most of the ESD results found in the literature are measurements of ejected ions. The principal reason for this is the ease of ion detection and the inherently low sensitivity of detectors to neutral particles. Most information pertaining to the relative abundance of neutral versus ion desorption is the result of indirect measurements. Despite extensive experimentation including the application of resonant enhanced multiphoton ionization for the selective detection of atomic oxygen, the production of atomic oxygen via ESD was never detected. From careful calibration experiments the upper limit to the neutral to ion yield, O/O^+, by ESD was calculated to be 10.

In addition to the main thrust of the research outlined above, during the final year of the project Dr. P. Xu worked on two very similar projects involving structure determination of clean surface reconstructions using either only ultrahigh vacuum transmission electron diffraction (UHV-TED) for Si(001)-2x1 or UHV-TED in combination with UHV-high resolution plan view transmission electron microscopy (UHV-HREM) for Ir(001)-5x1 surface. The goal of these investigations was to resolve the controversy that existed about the actual clean surface reconstruction structure for these two surfaces.

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Introduction

The research discussed in this report is part of a several year study of the electron stimulated desorption (ESD) behavior of transition metal oxides. The original objective of this research was the development and implementation of an ESD O atom source to be used in Low Earth Orbital (LEO) simulations of materials damage. Though the desorption of neutral particles has been widely postulated, most of the ESD results found in the literature are measurements of ejected ions. The principal reason for this is the ease of ion detection and the inherently low sensitivity of detectors to neutral particles. Most information pertaining to the relative abundance of neutral versus ion desorption is the result of indirect measurements. To date, only one research group has reported the detection of ESD generated oxygen atoms.[1] The authors indicate that the O/O^+ ratio is 1.1×10^7. Given the size of the O^+ signals observed in our experiments, and the quoted O/O^+ ratio, neutral oxygen should have been detected in our experiments, but was not detected. As the negative results accumulated in this study, the objective changed from using the ESD process for LEO simulation studies to studying the ESD process itself. This report describes the results of our experiments which clearly indicate that the ESD O/O^+ ratio, in general, is many orders of magnitude smaller than that claimed in Reference 1.

In addition to the main thrust of the research outlined above, during the final year of the project Dr. P. Xu worked on two very similar projects involving structure determination of clean surface reconstructions using either only ultrahigh vacuum transmission electron diffraction (UHV-TED) for Si(001)-2x1 or UHV-TED in combination with UHV-high resolution plan view transmission electron microscopy (UHV-HREM) for Ir(001)-5x1 surface. The goal of these

investigations was to resolve the controversy that existed about the actual clean surface reconstruction structure for these two surfaces.

EXPERIMENTAL RESULTS

A. Quadrupole Mass Spectrometry

After attempts to detect ESD atomic oxygen with either a UTI 100C or a VG Masslab quadrupole mass spectrometer (QMS) were unsuccessful because of interference from CH_4 at mass 16, it was determined that truly unambiguous results demanded the use of a high resolution, high performance QMS. To meet this need, an Extrel C-50 QMS was procured. This instrument provides total separation of O atoms (15.99 amu) from the residual methane (16.03 amu) typically found in UHV systems. With this instrument it was possible to monitor both signals independently, and an ESD induced change in either signal could be unambiguously confirmed.

It was observed, in general, that the firing of the electron gun caused an increase in many mass signals in addition to the ^{16}O signal. This could have suggested that ESD O atoms (as well as other products) were desorbed in the electron beam. The source of the ^{16}O increase was probed by rejecting the ESD oxygen ions with an ion repeller positioned between the target and the QMS, and analyzing the remaining neutral fraction with the appearance potential technique. The energy required to produce O^+ from O atoms, CO_2, and CO (13.6, 19.5, and 23.6 eV respectively) may be used to identify the neutral ^{16}O source by lowering the mass spectrometer's electron impact energy until the signal disappears. A result typical of those repeatedly observed in this project is the high resolution ESD spectrum from oxidized V shown in Fig. 1. The source of the neutral fraction is seen to have arisen from CO.

Another QMS technique used in this project was one in which ions of the same nominal mass are distinguished in the QMS on the basis of their kinetic energy. By increasing the pole bias ~3 V above ground potential, background gases and their fragments are filtered out by the quadrupole while the higher energy ESD particles are transmitted. The experiment was carried out on a flame oxidized V target and an $^{18}O_2$ oxidized Ag target as follows: First, the quadrupole resolution was lowered to increase the transmission of all species, with the expectation that the higher throughput would aid in detecting a small ESD neutral signal. The background spectrum is shown in Fig. 2. The pole bias was then raised by 3.5 V (Fig. 3) to filter the low energy mass 16 particles (CH_4 and ^{16}O from CO or CO_2). Note the large change in signal intensity. The ESD ion spectrum (Fig. 4) was collected with the ionizer turned off and the ion repeller at ground potential. The desorbate ions were then rejected by the application of ~+20 V to the ion repeller grid (result not shown). The QMS ionizer was then turned back on and the "neutral" ESD spectrum (Fig. 5) was collected. The benefit of using $^{18}O_2$ is that the target could be the only source of ^{18}O and any unwanted ESD $^{16}O^+$ generated *in the ionizer* cannot interfere with the neutral ESD signal. From Fig. 5 it is evident that there is no ESD ^{18}O to be seen.

This experiment points out an important experimental problem; the unavoidable production of ESD ions by the QMS's electron impact ionizer. After the pole bias was increased, a small mass 18 signal persists in Fig. 3. This signal was likely caused by a tiny fraction of energetic H_2O^+ produced by ESD inside the QMS ionizer. It was the inability to prevent the unwanted ESD of $^{16}O^+$ during $^{16}O_2$ oxidation experiments that forced the use of $^{18}O_2$. In cases where the desired signal is much larger than the background, this unintended ESD process is insignificant. In the detection of ESD generated neutral oxygen, however, this interference must

be carefully addressed. Unfortunately, it was found that all experimental efforts to minimize the unintended ESD signal served to minimize the desired signal as well. This pattern of results was observed throughout the QMS experiments. Despite the wide variety of materials and methods employed for the preparation of ESD target materials (Table 1) and despite the fact that O^+ has been detected from every ESD target examined, ESD atomic oxygen was never detected in this study. The QMS data analysis was concluded by calculating an upper limit to the ESD neutral to ion ratio based on the detection sensitivity of the QMS and the magnitude of the O^+ signal. Briefly, the Extrel's neutral detection limit is estimated from residual gas analyses with a signal to noise ratio of 10 measured at $\approx 3 \times 10^{-11}$ Torr. From the methane signal (<10% of background) the detection limit was determined to be $10^3 - 10^4$ cm^{-3}. Since the primary objective was the detection of 5 eV atomic oxygen, the detection threshold is expressed as the minimum 5 eV O atom flux required to produce the limiting neutral density. The result is a detection limit of $\approx 8.5 \times 10^8$ 5 eV O atoms cm^{-2} sec^{-1} at the mass spectrometer.

The upper limit of the ESD O/O^+ ratio from the oxidized Ag target, for example, is calculated as follows: from Table 2 the O^+ signal recorded for Ag is 1.0×10^5 sec^{-1}. The ion count rate is converted to an ion flux at the mass spectrometer by including the quadrupole transmission factor and the face plate aperture size. The result is 3.0×10^7 O^+ cm^{-2} sec^{-1}. This means that based on the calculated detection sensitivity of the QMS to 5 eV O atoms, an upper limit to the ESD O/O^+ ratio is ≈ 28. The other limits presented in Table 2 were calculated in the same way.

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B. X-Ray Photoelectron Spectroscopy

In addition to the QMS experiments just described, the surface of an oxidized V foil was analyzed with XPS then subjected to 150 minutes of electron irradiation (850 V, 150 μA beam current), then reanalyzed with XPS to determine the extent of ESD induced oxygen loss. The "total" oxygen loss was compared to the measured O^+ yield to determine the degree to which the oxygen depletion could be attributed to ESD of O^+ and hence calculate the ESD O/O^+ ratio.

Fig. 6 shows the shift in the V 2p peaks resulting from electron beam induced reduction of the oxide. The shift is toward lower binding energy and results from reduction of the surface. The changes in the magnitude of the V peaks before (solid line) and after (dashed line) irradiation are not of concern here. Rather the quantity of interest is the change in the O/V peak ratios before and after irradiation. Following 2.5 hours of irradiation this ratio was found to decrease by ≈10%.

The O atom concentration in the target, before irradiation, was calculated by assuming that the surface of the oxidized V foil is initially stoichiometric V_2O_5. From the V_2O_5 unit cell dimensions, it is seen that there are 1.22×10^{15} O atoms cm^{-2} with 4.37 Å between atomic layers. The number of layers probed during the XPS analysis is limited by the inelastic mean free path of the escaping photoelectron. From the photoelectron kinetic energy, x-ray photon energy, and the inelastic mean-free-path the signal is seen to arise from the top two atomic layers. With this information, it was determined that the effect of 2.5 hours of electron irradiation upon the oxidized V target, was the loss of 2.44×10^{14} O species cm^{-2}.

Based upon ESD experiments carried out on other V foil targets and the electron gun output during the 2.5 hours, the integrated ESD O^+ yield, corrected for the transmission, solid

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angle, and electron beam area is 2.75×10^{13} O^+ cm^{-2} for the 150 minutes of irradiation. If it is assumed that all unaccounted oxygen loss, as measured by the XPS, is attributable to O atom desorption, then the upper limit of the ESD O/O^+ ratio is ~9.

C. Resonance Enhance Multiphoton Ionization-Time-Of-Flight-Mass Spectrometry

In light of the difficulty in detecting fast moving particles with a conventional mass spectrometer using an electron impact ionizer, an alternative method was employed based on laser ionization of atomic oxygen. The frequency doubled output of an excimer pumped dye laser was used to ionize the ESD O atoms in a 2+1 resonance enhanced multiphoton ionization (REMPI) process at 226 nm with an ionization efficiency of 1-10%. A time-of-flight mass spectrometer was employed for mass-resolved detection of the photoionized O atoms. This set up allowed the detection of ESD ions and neutrals thereby enabling us to measure the O/O^+ flux ratio and both the O and O^+ kinetic energies. For reference, a representation of the ESD-MPI-TOF system is shown in Fig. 7.

Fig. 8 shows an ESD-TOF ion spectrum collected from a flame oxidized Ag foil target. With the target held at ground potential (8a), a large ESD O^+ signal was recorded near 11 μsec flight time. The ion signal was suppressed by application of 10 V (8b) to establish a baseline in preparation for the laser probe of neutral ESD products. The results of the neutral probe are displayed in Fig. 9. Fig. 9b is typical of the "neutral" ESD-MPI-TOF spectra observed in this study. The spectrum consists of a single, prominent, laser dependent feature at ~13.3 μsec. Its identity is confirmed by comparison to Fig. 9a, a gas phase photodissociation, photoionization spectrum of NO_2. The neutral peak in Fig. 9a. arose from residual NO which was a significant

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contaminant in the NO_2 used to optimize the apparatus voltages, wavelength... shortly before Fig. 9b was recorded.

The detection limit for the MPI-TOF apparatus and an upper limit to the ESD O/O^+ ratio were computed. From calibration experiments using NO_2 it was determined that the minimum partial pressure of NO_2 required to give a detectable O atom MPI signal was 9.6 x 10^{-12} Torr. This corresponds to an O atom concentration of 3.4 x 10^5 cm^{-3} or a minimum detectable 5 eV O atom flux of 2.6 x 10^{11} cm^{-2} sec^{-1} at the laser focal point which corresponds to a flux at the surface of 5.2 x 10^{13} cm^{-2} sec^{-1}. The ESD O^+ flux from the Ag target was determined to be 6.1 x 10^{12} by integrating a spectrum such as that shown in Fig. 8. The result is an upper limit to the ESD O/O^+ ratio of =8.5. That is to say that there must be less than 10 oxygen atoms produced for every oxygen ion produced, for otherwise an ESD atomic oxygen signal would have been detected and none was ever detected.

D. Surface Structure of Ir(001) and Si(001)

Both the Ir(001) and Si(001) samples were prepared by a combination of mechanical polishing, dimpling and ion milling external to the ultrahigh vacuum (UHV) transmission electron microscope facility (till a small hole was obtained at the center of the sample). These were then subjected to a series of ion milling and annealing cycles inside the UHV-surface science chamber (UHV-SSC) attached to the UHV-TEM till clean reconstructed surfaces could be obtained. In case of Ir(001),[2] the changes in surface morphology, in particular the appearance and evolution of steps, were characterized as a function of the milling and annealing treatments. A series of clean reconstructed surface diffraction patterns at different exposure times were recorded in both systems. These were then digitized and the absolute intensity values were obtained using two

different approaches: a) by removing the background using a high pass filter and then integrating the intensity around each spot as was used for Ir(001)-5x1.[3] b) In case of Si(001)-2x1 system analysis, a better approach i.e. a cross-correlation technique[4] was used for the same purpose. The intensities of the bulk spots were used in numerical multislice calculations to establish the tilt and thickness conditions. HREM images were used in Ir(001)-5x1 system to rule out other exotic structures for the surface. The intensities corresponding to the contradicting structure models for both systems were calculated using a rigorous multislice dynamical approach and the structure that gave the least deviation i.e. minimum value of the reliability factor (R-factor) was then proposed as the clean surface atomic structure. In case of Si(001)-2x1 surface,[5] since the structure could not be resolved solely on the basis of tne value of the R-factor alone, an error estimate i.e. a reduced χ^2 fit was also carried out (this level of sophistication had not been attained at the time of the Ir(001)-5x1 analysis).

CONCLUSION

The ESD process from the oxides of five transition metals has been studied using high resolution quadrupole mass spectrometry, X-ray photoelectron spectroscopy, and multiphoton ionization-time-of-flight mass spectrometry to determine whether many neutral particles are produced for every ion produced as has been recently reported. From the QMS results it was determined that the ESD O/O^+ ratio, from all samples examined is, on average, not more than 125. The XPS study on oxidized V sets this ratio at less than 10. From the MPI experiments on oxidized Ag the ratio also appears to be less than ten. The principal conclusions drawn from these experiments are that the ESD process is not viable manner in which to produce atomic

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oxygen and that the O/O^+ ratio is several orders of magnitude smaller than the value of 10^7 claimed in reference 1.

PERSONNEL

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                                        Professor Laurence D. Marks
                                        Professor David Seidman
                                        Professor Eric Weitz

Postdoctoral Associates: Dr. F. J. Northrup and Dr. P. Xu

Graduate Students: Mitch Jacoby

Publications

(1)   "An Investigation of the ESD O/O^+ Ratio from V_2O_5 and Oxidized V", Mitch Jacoby, F. J. Northrup, P. C. Stair, E. Weitz, and L. D. Marks, in preparation

(2)   "The Electron Stimulated Desorption of Oxygen from Oxidized Ag", Mitch Jacoby, F. J. Northrup, P. C. Stair, E. Weitz, and L. D. Marks, in preparation

Presentations   .

"Radiation Damage in Transition Metal Oxides"
Amoco Research Center Poster Presentations, Naperville, October 1991.

"The Electron Stimulated Desorption of Oxygen from Transition Metal Oxides"
DIET V, 5th International Conference On Desorption Induced by Electronic Transitions
Taos, New Mexico, April 1992.

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3.   L. D. Marks, P. Xu and D. N. Dunn, *Surf. Sci.* **294**, 322 (1993).

4.   P. Xu, G. Jayaram and L. D. Marks, *Ultramicroscopy* (In Press).

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TABLE 1

ESD TARGET MATERIALS AND PREPARATION METHODS

ELEMENT   **PREPARATION METHOD**

SILVER   foil, 99.99% (Aldrich), 0.125 mm thick,   heated to 500°C in 1 atm. $^{16}O_2$.

    "   "   "   heated to 500°C in 1 atm. $^{18}O_2$.

    "   "   "   dosed with O_2, 1 atm., 25°C.

    "   "   "   heated in flame to incandescence.

    "   0.250 mm thick,   flame oxidized.

TANTALUM   foil, 99.7% (Aldrich), 0.025 mm thick, heated in flame to incandescence.

TITANIUM   foil, 99.7% (Alfa Products), 0.025 mm thick,
                                                heated in flame to incandescence.
               TiO_2 powder, 99.998 (Cerac), pressed pellet, sintered.

TUNGSTEN   foil, 99.9% (Aldrich), 0.05 mm thick, heated in flame to incandescence.

               foil, 99.9% (Aldrich), 0.05 mm thick, dosed with O_2, 12 psi., 25°C.

               WO_3 powder, 99.995% (Aldrich) pressed into In foil.

VANADIUM V_2O_5 powder, 99.9% (Aesar), pressed pellet, untreated.

    "   "   heated to 550°C in 1 atm.O_2.

    "   "   sintered (625°C, 1 atm O_2, 12hr).

    "   " H_2 reduction + ^{18}O oxidation.

               foil, 99.7% (Aldrich), 0.127 mm thick, heated in flame to incandescence.

TABLE 2

UPPER LIMITS of the ESD O/O$^+$ FLUX RATIO

TARGET MATERIAL	MAXIMUM O$^+$ SIGNAL (counts/second)	$\Phi_{QMS} O^+$ (O$^+$cm^{-2}sec^{-1})	O/O$^+$ RATIO
OXIDIZED Ag FOIL	1.0×10^5	3.0×10^7	28
OXIDIZED Ta FOIL	4.0×10^4	1.2×10^7	70
OXIDIZED Ti FOIL	4.0×10^4	1.2×10^7	70
OXIDIZED V FOIL	2.4×10^4	7.3×10^6	117
OXIDIZED W FOIL	2.0×10^4	6.1×10^6	140
V$_2$O$_5$ PELLET	8.0×10^3	2.4×10^6	351
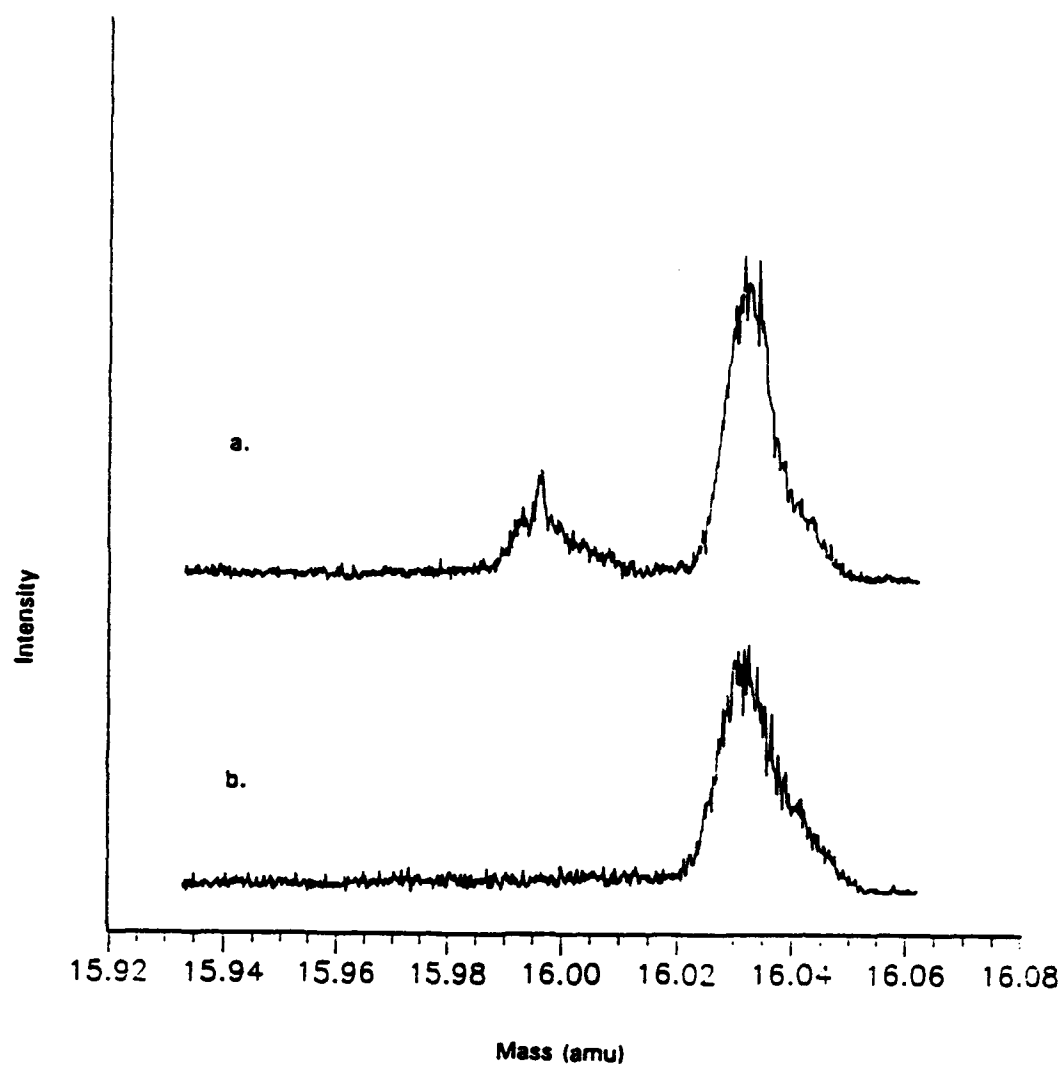
Figure 1.

Figure L.   Appearance potential analysis of the source of the ^{16}O neutral
fraction. (a) e- impact energy = 50 eV. (b) e- impact energy
= 23 eV.

Figure 2.

Figure 3.

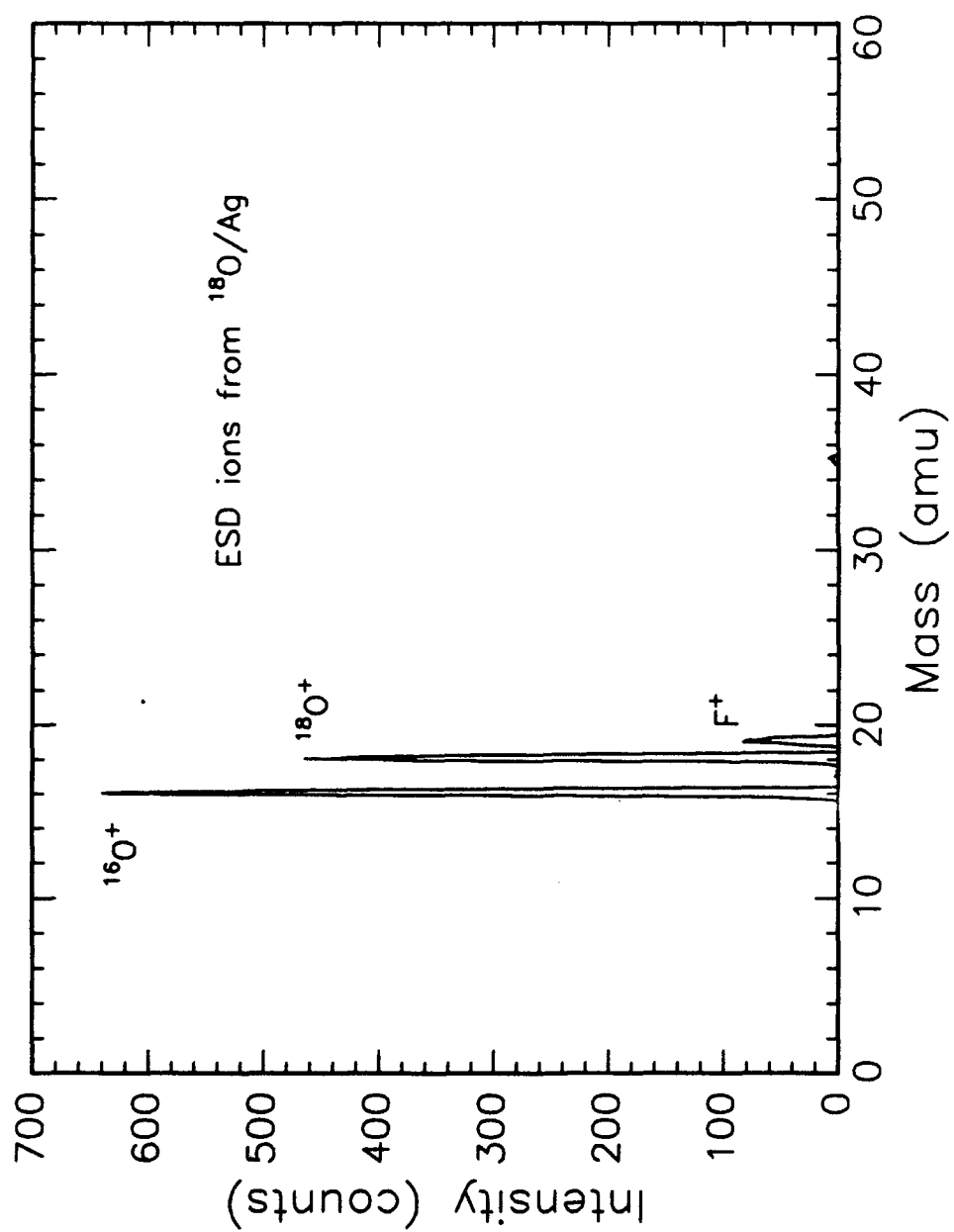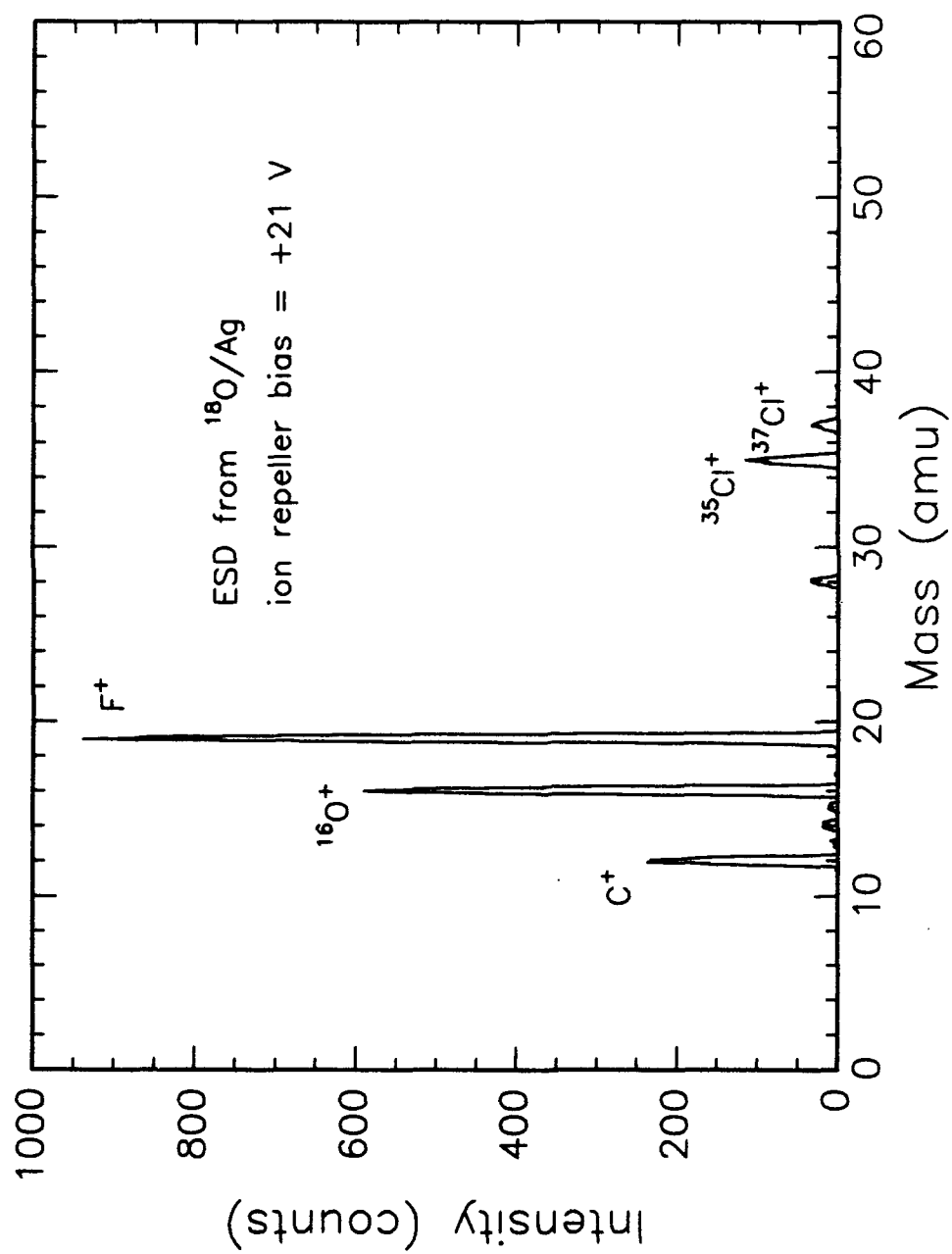RGA: pole bias = +3.5 V

Figure 4.

ESD ions from $^{18}O/Ag$

$^{16}O^+$

$^{18}O^+$

F^+

Mass (amu)

Intensity (counts)

Figure 5.

ESD from ^{18}O/Ag
ion repeller bias = +21 V

F^+

$^{16}O^+$

C^+

$^{35}Cl^+$

$^{37}Cl^+$

Mass (amu)

Intensity (counts)
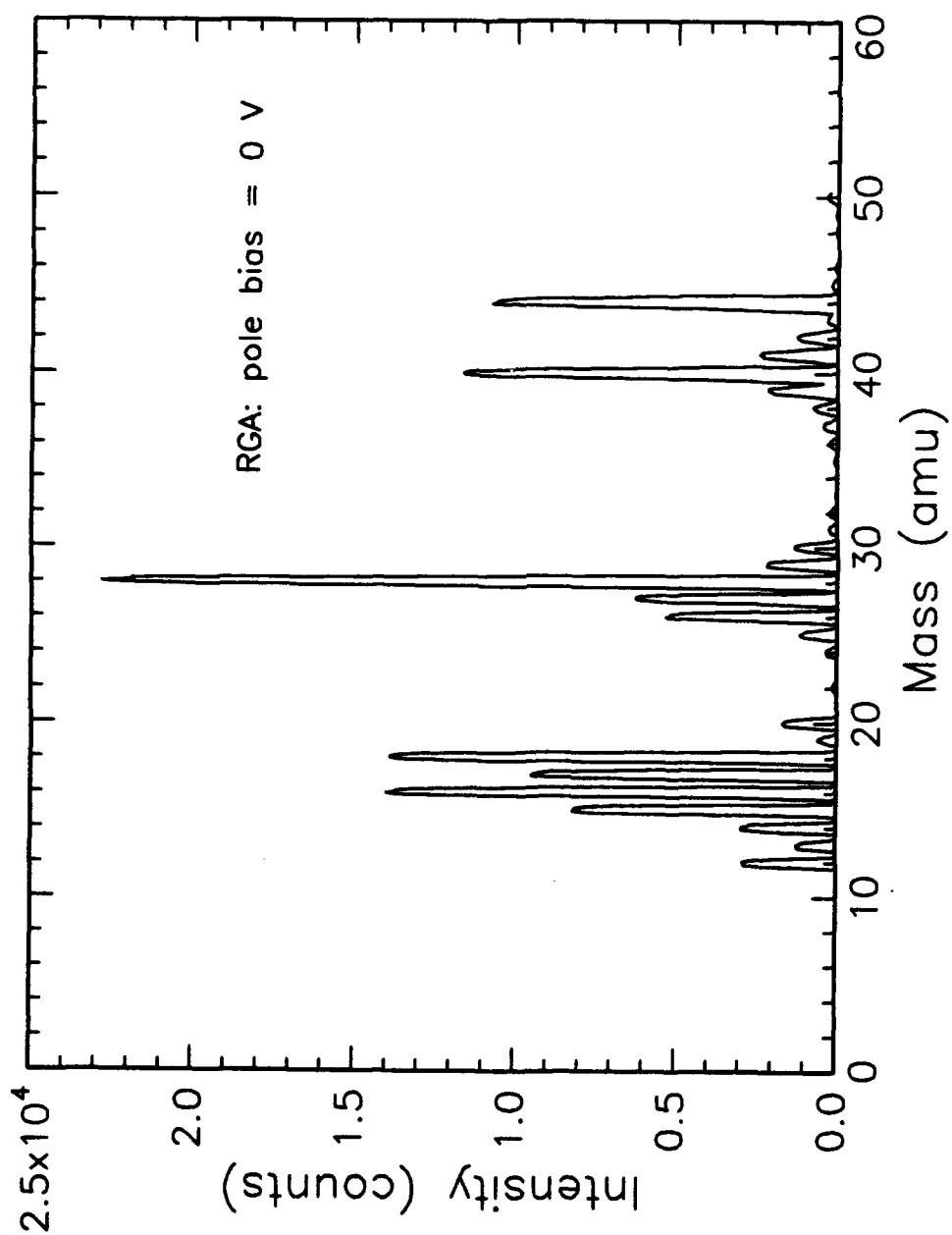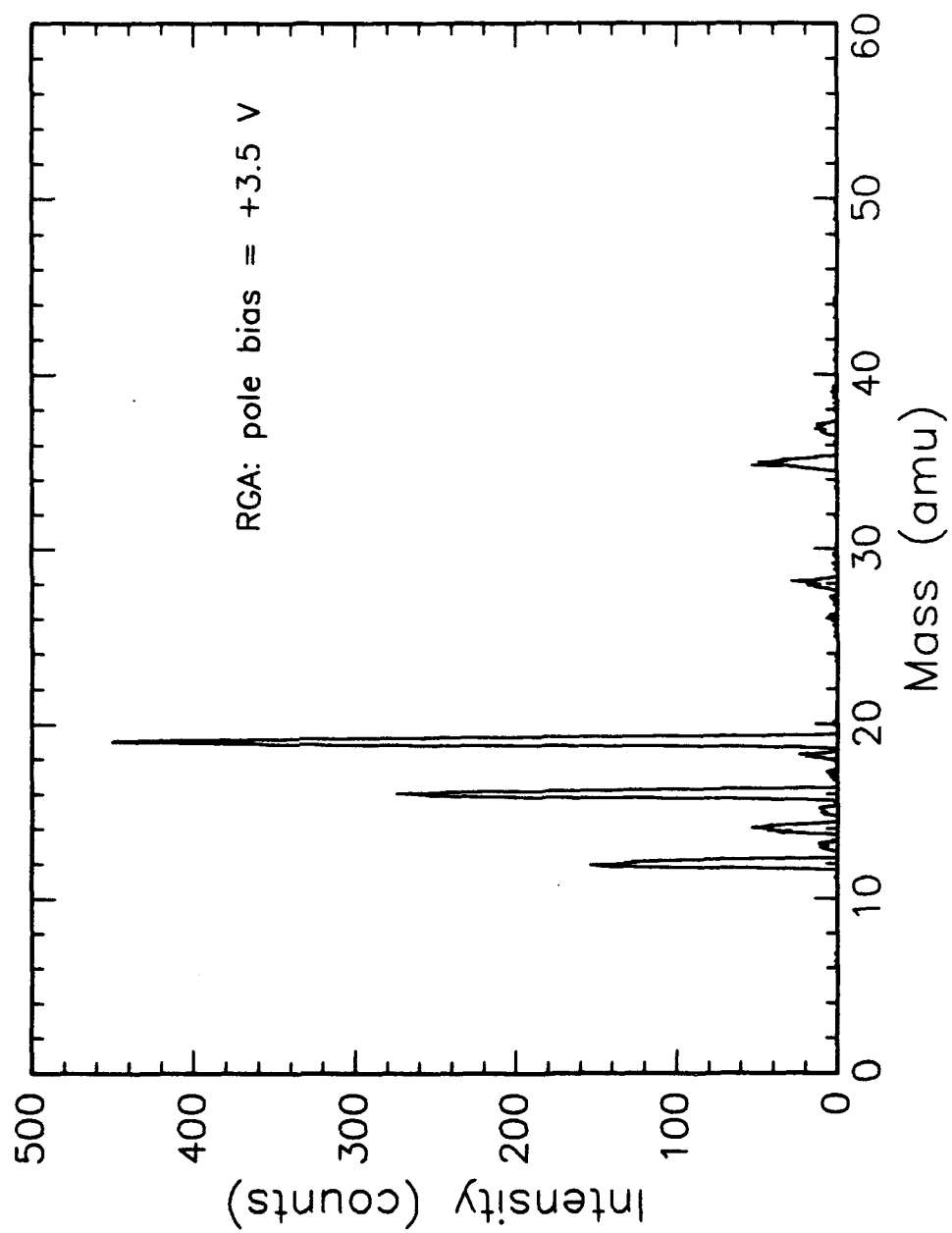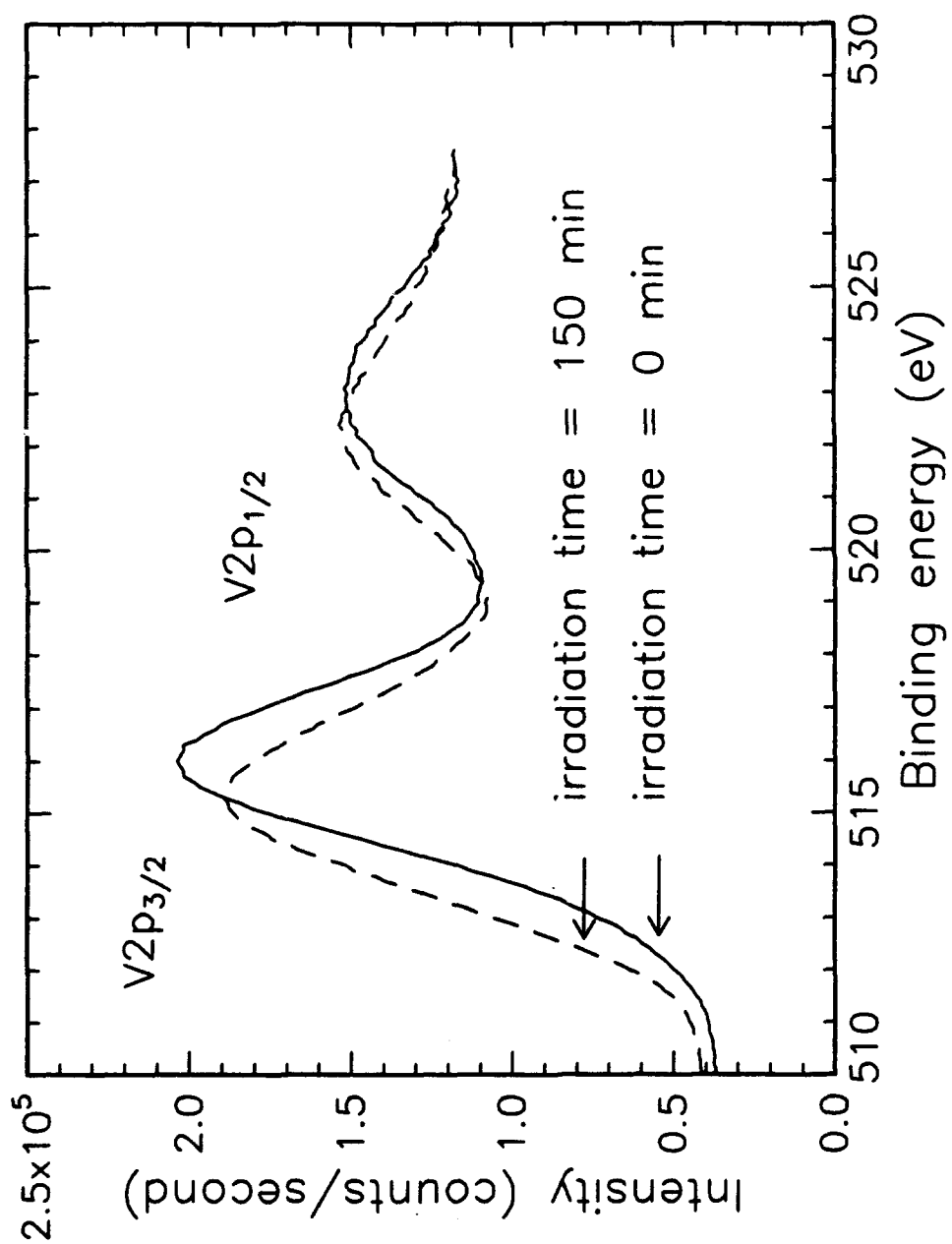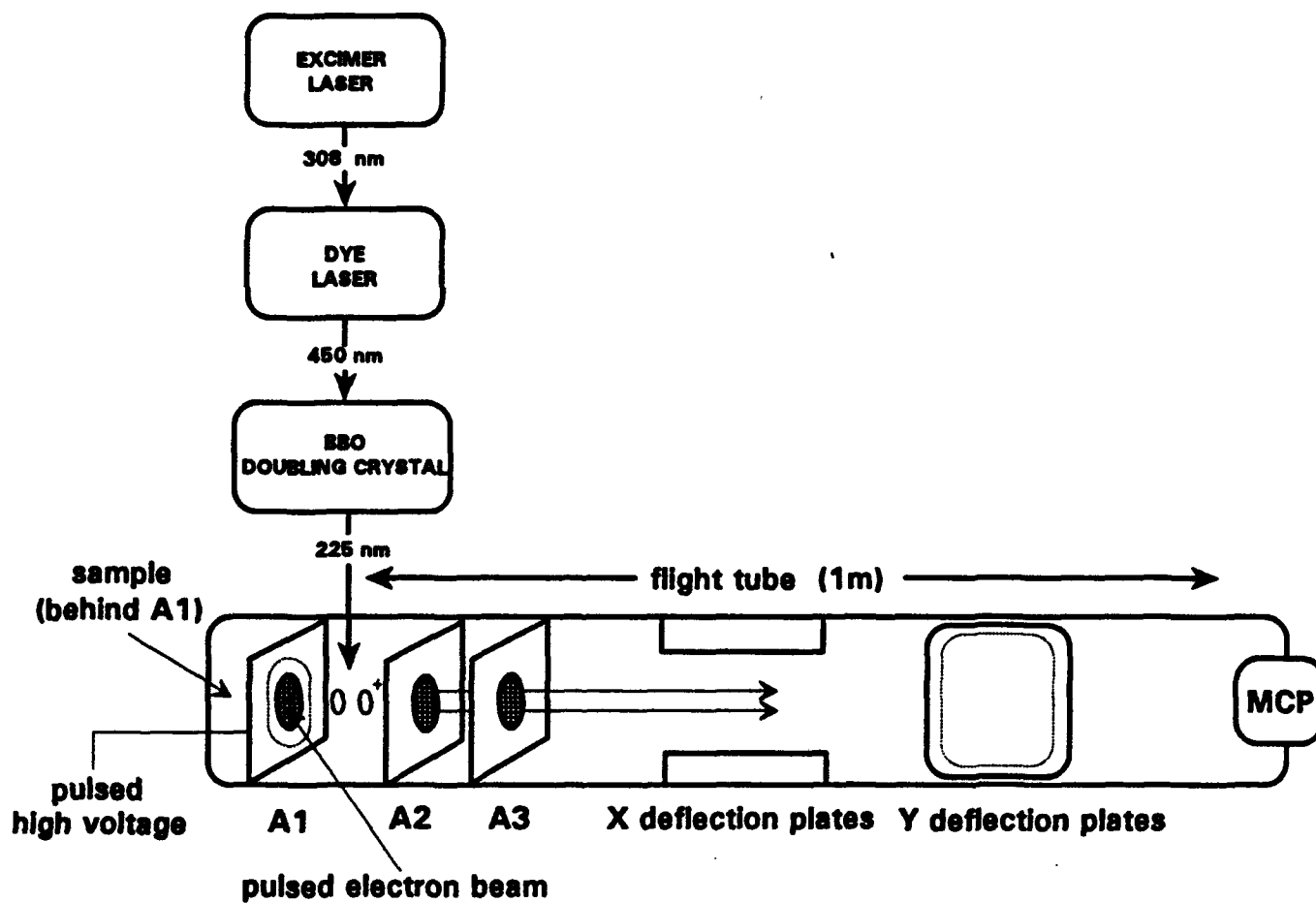
Figure 6.

Figure 7.

Figure 7. Experimental apparatus for the multiphoton ionization and time-of-flight detection of ESD generated atomic oxygen.
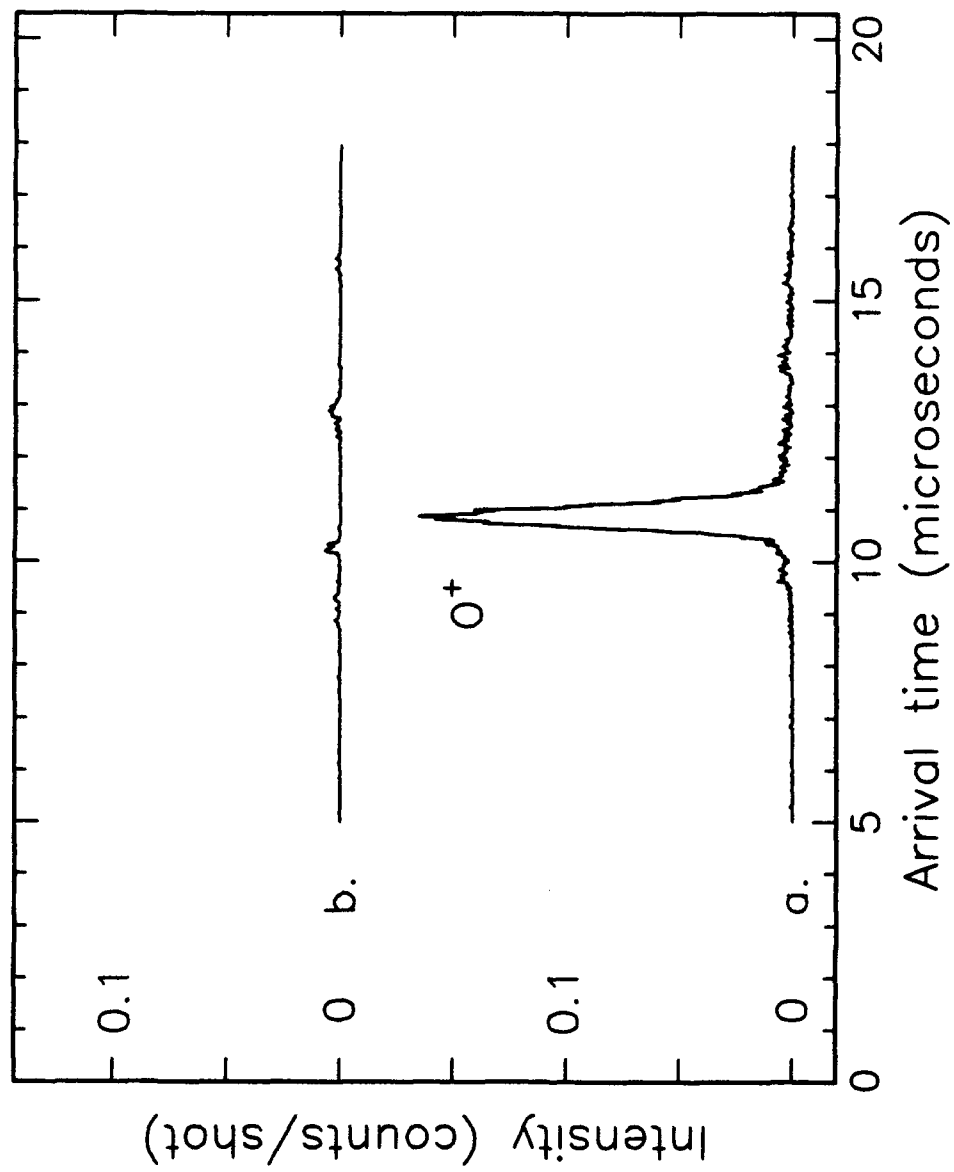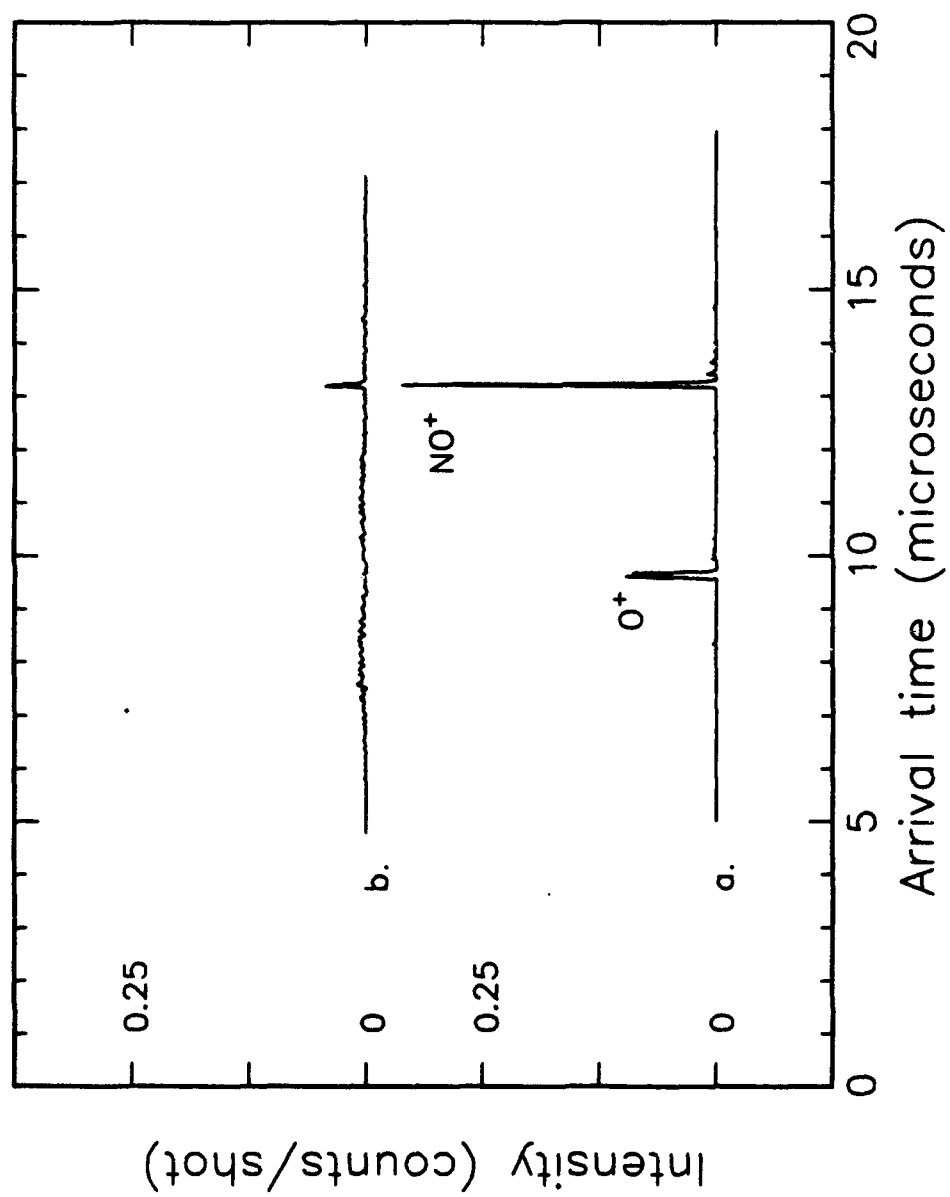
Figure 8.

Figure axes: Intensity (counts/shot) vs. Arrival time (microseconds), showing traces labeled b. and a. with an O^+ peak near 11 microseconds.

Figure 9.

COMPLETED PROJECT SUMMARY

TITLE: Experimental and Theoretical Investigation of Surface Chemistry Induced by Direct and Indirect Electronic Excitation

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